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A revised analytical method for stable Mo and W isotopes in sediments: implications for paleoenvironmental reconstruction

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Mo and W concentrations and their stable isotope ratios are widely used palaeoceanographic proxies; however, their analysis remains challenging because of their low concentrations in sediments. Tsujisaka et al. (2019) reported a method based on chemical separation by NOBIAS Chelate PA1 chelating resin column extraction and AG1-X8 anion-exchange resin chromatography, followed by measurement using MC-ICP-MS. This study improved the method in terms of operability and precision, evaluated its performance through analyses of geological reference materials including marine sediments (JMS1, JMS2), igneous rocks (JA3, JB3), and a manganese nodule (JMn1), and applied it to sediments from the Black Sea.

The major improvements are as follows. After evaporation of the HF–HNO_3–H_2O_2 mixture used for sample decomposition, the residues were dissolved in 0.06 M HCl (pH1.2–1.4), thereby eliminating the need for additional pH adjustment prior to column extraction using TSK-8HQ chelating resin. After sample loading, 200 mL of 0.06 M HNO_3 was passed through the column to remove Fe, and Mo and W were subsequently eluted with 5 M HF. The AG1-X8 anion-exchange resin column was downsized to a bed height of 2 cm, which reduced both eluent volumes and operation time. Residual Fe was removed using 0.5 M HF–0.4 M HCl instead of 1 M HF to prevent the loss of Mo and W. Evaporation temperature of the tungsten fraction was optimized to 130 °C to avoid the formation of tungsten oxides. As a result, one cycle of the chemical separation required ~ 11 h, compared with ~ 24 h for the previous method.

The Mo and W concentrations and their stable isotope ratios measured in this study were consistent with those reported in Tsujisaka et al. (2019). When Mo (Wako 130-14961) and W (Wako 205-20951) standard solutions were added to geological reference materials, quantitative recoveries of 100 ± 5% were achieved for both Mo and W. The regression intercepts for δ^{98}Mo (0.14–0.23‰) and δ^{186}W (0.04–0.09‰) obtained from the δ Mo vs 1/Mo and δ W vs 1/W plots were consistent with those of standard solutions.

Tsujisaka M. et al. (2019) Anal. Chim. Acta 1091, 146–159.